DT15 Rec'd PCT/PTO 23 SEP 2004

Specification

HIGH FLAME RESISTANT UNION FABRIC

#### FIELD OF THE INVENTION

The present invention relates to a flame resistant union fabric. Specifically, the present invention relates to a union fabric having high degree of flame resistance consisting of a compound yarn having a halogen-containing flame resistant fiber including an antimony compound as a principal component, and a cellulosic fiber.

#### 10 BACKGROUND ART

5

15

20

25

30

In recent years, demand for guarantee of safety of foods, clothes and housings has become stronger, and necessity for fire-resistant materials is increasing. In such a situation, a plurality of methods to give flame resistance to a flammable yarn by compounding general-purpose flammable fibers and flame resistant fibers having high degree of flame resistance, while maintaining characteristics of the flammable yarn, have been proposed. As such a compound fiber, for example, Japanese Patent No. 2593985 specification and Japanese Patent No. 2593986 specification disclose a method of using antimony compounds as flame resistant agent to be added to flame resistant halogen-containing fibers in compounding halogen-containing flame resistant fibers and natural fibers.

Recently, union fabrics using general-purpose cellulosic fibers as a warp yarn and a halogen-containing flame resistant fiber including antimony compounds as a weft yarn are often used for interior design products, such as curtains and chair coverings, because special features of cellulosic fibers, such as natural feeling, hygroscopic property, and heat resistance, can be exhibited. Among them, union fabrics using cellulosic fibers as a warp yarn and halogen-containing flame resistant fibers including antimony compounds as a weft yarn,

such as jacquard, dobby, and satin have special feature with many cellulosic fibers disposed on a surface side of the fabric.

However, in these union fabrics, uneven existence of cellulosic fibers and halogen-containing flame resistant fibers in a fabric makes it very difficult to pass a highest flame resistant class M1 in NF P 92-503 combustion test in France that requires a very high degree of flame resistance.

Only international publication No. 01/32968 pamphlet proposes a method applying such technique furthermore in which a union fabric using a cellulosic fiber as a warp yarn and a halogen-containing fiber having an antimony compound and a zinc stannate compound added therein in combination as a weft yarn has a very high flame resistance passing Class M1 of NF P 92-503 combustion test.

However, since zinc stannate compounds a have higher cost than that of antimony compounds, the fiber has a cost higher than that of conventional fibers as compared with independent addition of the antimony compounds to the halogen-containing fiber, leading to a problem of higher cost of the union fabric.

Accordingly, in a union fabric comprising a halogen-containing fiber by addition of only antimony compounds and a general-purpose fiber, such as a cellulosic fiber, development of a union fabric exhibiting high flame resistance and classified in Class M1 of NF P 92-503 combustion test without combined use of zinc stannate compounds etc. has been long awaited.

The present invention aims at providing a fabric having high degree of flame resistance in case of union fabrics consisting of halogen-containing flame resistant fibers and cellulosic fibers, and classified in class M1 of NF P 92-503 combustion test.

#### SUMMARY OF THE INVENTION

5

10

15

20

25

30

The present inventors performed repeated investigation about

union fabrics consisting of modacrylic flame resistant fibers as halogen-containing flame resistant fibers, and cellulosic fibers. As a result, it was found out that when a compound yarn using a modacrylic fiber, compounded with other fibers, including antimony compound as a principal component shows a certain specific thermal behavior, use of the compound yarn as a warp yarn or a weft yarn might exhibit high flame resistance in union fabrics, such as jacquard, dobby, and satin weave.

That is, the present invention relates to a flame resistant union fabric obtained by co-weaving: a compound yarn (A) 30% to 70% by weight obtained by compounding a halogen-containing flame resistant fiber (a-1) including an antimony compound 25 parts (hereinafter abbreviated as simply part) to 50 parts into an acrylic based copolymer 100 parts obtained by copolymerizing a monomer mixture comprising acrylonitrile 30% to 70% by weight (hereinafter abbreviated as simply %), a halogen containing vinyl based monomer 30% to 70%, and a vinyl based monomer copolymerizable therewith 0% to 10%, with an other fiber (a-2), the compound yarn (A) having less than 5% of elongation under a condition of a load of 300 mg/metric count of No. 17, and of a temperature range of 100 degrees C to 500 degrees C; and a cellulosic fiber yarn (B) 70% to 30% by weight.

The flame resistant union fabric is preferably of a union fabric wherein the cellulosic fiber (B) is at least one kind selected from a group consisting of cotton, hemp, rayon, polynosic, cupra, acetate, and triacetate.

### BEST MODE FOR CARRYING-OUT THE INVENTION

5

10

15

20

25

30

The present invention relates a flame resistant union fabric obtained by compounding:

a compound yarn (A) 30% to 70% by weight obtained by compounding a halogen-containing flame resistant fiber (a-1) including an antimony

compound 25 parts to 50 parts into an acrylic based copolymer 100 parts obtained by copolymerizing a monomer mixture comprising acrylonitrile 30% to 70%, a halogen containing vinyl based monomer 30% to 70%, and a vinyl based monomer copolymerizable therewith 0% to 10%, with an other fiber (a-2), the compound yarn (A) having less than 5% of elongation under a condition of a load of 300 mg/metric count of No. 17, and of a temperature range of 100 degrees C to 500 degrees C; and a cellulosic fiber yarn (B) 70% to 30% by weight.

5

10

15

20

25

30

the present invention, a fiber yarn including halogen-containing flame resistant fiber (a-1) is a fiber used in order to give flame resistance to a union fabric of the present invention. The halogen-containing flame resistant fiber (a-1) consists of a composition including an antimony compound in an acrylic based copolymer obtained by polymerizing a monomer mixture including acrylonitrile 30 to 70%, halogen containing vinyl based monomer 30% to 70%, and a vinyl based monomer copolymerizable with the acrylonitrile and the halogen containing vinyl based monomer (hereinafter referred to as copolymerizable vinyl based monomer) 0% to 10%.

In the monomer mixture used for obtaining the acrylic based copolymer, a percentage of the acrylonitrile is not less than 30%, and preferably not less than 40% (lower limit), and it is not more than 70%, and preferably not more than 60% (upper limit).

In the monomer mixture, a percentage of the halogen containing vinyl based monomer is not less than 30%, and preferably not less than 40% (lower limit), and it is not more than 70%, and preferably not more than 60% (upper limit).

In the monomer mixture, a percentage of the copolymerizable vinyl based monomer is preferably not less than 1% (lower limit), and it is not more than 10%, and preferably not more than 5% (upper limit).

Of course, a total percentage of the acrylonitrile, the halogen containing vinyl based monomer, and the copolymerizable vinyl based monomer is adjusted so as to give 100%.

5

10

15

20

25

30

In the monomer mixture, a percentage of the acrylonitrile of less than the lower limit or a percentage exceeding the upper limit of the halogen containing vinyl based monomer does not allow demonstration of sufficient heat-resistance, and a percentage exceeding the upper limit of the acrylonitrile unit or a percentage of the halogen containing vinyl based monomer of less than the lower limit gives inadequate flame resistance. In the monomer mixture, a percentage exceeding the upper limit of the copolymerizable vinyl based monomer fails to fully exhibit flame resistance and touch that are special feature of the halogen-containing flame resistant fiber.

Any halogen containing vinyl based monomers can be used, as long as the halogen containing vinyl based monomer is a vinyl based monomer including halogen atom, preferably bromine atom or chlorine atom. As examples of the halogen containing vinyl based monomer, for example, vinyl chloride, vinylidene chloride, vinyl bromide, etc. may be mentioned. These may be used independently or two or more kinds may be used in combination.

As the copolymerizable vinyl based monomer, for example, there may be mentioned: acrylic acid; acrylic esters, such as ethyl acrylate, and propyl acrylate; methacrylic acid; methacrylic esters, such as methyl methacrylate, and ethyl methacrylate; and furthermore, acrylamide, vinyl acetate, vinyl sulfonic acid, vinyl sulfonate (sodium vinyl sulfonate etc.), styrene sulfonic acid, styrene sulfonate (sodium styrene sulfonate etc.) These may be used independently or two or more kinds may be used in combination.

As methods of obtaining the acrylic based copolymer by polymerization of the monomer mixture including the acrylonitrile,

halogen containing monomer, and the monomer copolymerizable therewith, any methods, such as usual vinyl polymerization methods, for example, a slurry polymerization method, an emulsion polymerization method, a solution polymerization method, etc., may be adopted without special limitation.

As preferable examples of the antimony compound, for example, inorganic antimony compounds, such as antimony trioxide, antimony pentoxide, antimonic acid, and antimony oxychloride may be mentioned. These may be used independently or two or more kinds may be used in combination.

A content of the antimony compound is not less than 25 parts to the acrylic based copolymers 100 parts, and preferably not less than 30 parts (lower limit), and it is not more than 50 parts (upper limit). A content of the antimony compound of less than the lower limit disables sufficient guarantee of flame resistance of a compounded flame resistant union fabric. And on the other hand, an amount the antimony compound exceeding the upper limit reduces physical properties, such as strength and elongation, of the halogen-containing flame resistant fiber, leading to problems, such as nozzle clogging during manufacturing process.

As methods of adding the antimony compound, as a flame resistant agent, to the acrylic based copolymer to obtain a composition (halogen-containing flame resistant fiber), there may be mentioned: a method of dissolving the acrylic based copolymer in a solvent that can dissolve the copolymer and then of mixing and dispersing the flame resistant agent into the obtained solution to manufacture a fiber; and a method of immersing a fiber obtained from the acrylic based copolymer into a binder aqueous solution including a flame resistant agent and then squeezing, drying, and heat treating to impregnate the flame resistant agent using after treatment technique etc. Methods

for obtaining a halogen-containing flame resistant fiber are not limited to them, and other well-known methods may be used.

As long as a compound yarn (A) obtained by compounding a halogen-containing flame resistant fiber (a-1) and an other fiber (a-2) is a compound yarn having less than 5% of elongation percentage under conditions of a load of 300 mg/metric count of No. 17 and of a temperature range of 100 degrees C to 500 degrees C, the other fiber (a-2) compounded with the halogen-containing flame resistant fiber (a-1) is not especially limited. An elongation percentage is more preferably not more than 3%. Since not less than 5% of elongation percentage of the compound yarn (A) reduces heat resistance and flame resistance, leading to formation of a hole in a textile fabric when ignited.

5

10

15

20

25

30

Here, an elongation percentage of the compound yarn (A) is obtained by measuring a sample length under a fixed load of 300 mg/metric count of No. 17, when a temperature is raised from 100 degrees C to 500 degrees C at a rate of 100-degree C/minute using SSC150 (manufactured by Seiko Instruments Inc.) An elongation percentage is defined as a ratio of a difference between a sample length at the time of a maximum elongation at 100 degrees C to 500 degrees C, and an original sample length, with respect to an original sample length.

Since a compound yarn (A) having an elongation percentage less than 5% of thermal behavior under conditions of a load of 300 mg/metric count of No. 17, and of a temperature range of 100 degrees C to 500 degrees C may be obtained, cotton, rayon, aramid fibers, nylon fibers, etc. are preferable as the other fiber (a-2). Since especially natural touch of the fabric can fully be exhibited, cotton and rayon are preferable.

A percentage of the halogen-containing flame resistant fiber (a-1) is preferably 60 parts to 95 parts in the compound yarn (A),

and more preferably 70 parts to 80 parts. And a percentage of the other fiber (a-2) is preferably 40 parts to 5 parts in the compound yarn (A), and more preferably 30 parts to 20 parts. The halogen-containing flame resistant fiber (a-1) and the other fiber (a-2) are compounded so as to be 100 parts in total.

5

10

15

20

25

30

There is shown a tendency for an amount of the halogen-containing flame resistant fiber (a-1) of less than 60 parts to reduce a content of the halogen-containing fiber exhibiting flame resistance in the fabric, resulting in decrease in flame resistance. There is also shown a tendency for an amount exceeding 95 parts of the halogen-containing flame resistant fiber (a-1) to melt the compound yarn (A) to easily form a hole in the fabric during combustion test, also resulting in decrease in flame resistance.

Compounding methods of the halogen-containing flame resistant fiber (a-1) and the other fiber (a-2) are not especially limited, and blending, twisting, etc. may be mentioned.

The cellulosic fiber yarn (B) can be used without special limitation. As examples, in view of fully exhibiting natural touch, at least one kind of yarns selected from a group consisting of cotton, hemp, rayon, polynosic, cupra, acetate, and triacetate are preferable. In view of many advantages, such as washing resistance, dye affinity, and low cost, especially cotton is preferable among them.

A flame resistant union fabric of the present invention is manufactured by co-weaving of the compound yarn (A) and the cellulosic fiber yarn (B) for giving heat-resistance and natural touch.

The flame resistant union fabric of the present invention is obtained by co-weaving one of the compound yarn (A) and the cellulosic fiber yarn (B) for a warp yarn, and another for a weft yarn, respectively.

Union fabric itself is a fabric excellent in design having very

characteristic appearance, and especially in co-weaving of the flame resistant fiber and general non-flame resistant fibers, some certain weaving methods enable a large amount of disposition on a fabric surface of non-flame resistant fibers with excellent touch or hygroscopic property, enabling increase in commercial value of the fabric. However, union fabrics that dispose much non-flame resistant fibers to a fabric surface thereof have low flame resistance in general as compared with that of plain fabrics. A union fabric of the present invention obtained by co-weaving the compound yarn (A) and the cellulosic fiber yarn (B) uses the compound yarn (A) obtained by compounding the halogen-containing flame resistant fiber (a-1) and the other fiber (a-2), and thereby while maintaining high degree of flame resistance of class M1 also in a union fabric, the union fabric allows disposition of a large amount of cotton (B) in the fabric surface, enabling realization of a fabric having high design property, excellent touch, and excellent hygroscopic property. It the union fabric, compounding of not only the halogen-containing flame resistant fiber but the other fiber (a-2) as the compound yarn (A) may suppress contraction by heat, promotes carbonization, and improves flame resistance. Furthermore, both of special features such as flame resistance of the compound yarn (A), and touch of the cellulosic fiber yarn (B) will be maximized.

5

10

15

20

25

30

In the flame resistant union fabric, a percentage of the compound yarn (A) is not less than 30%, and preferably not less than 40% (lower limit), and not more than 70%, and preferably not more than 60% (upper limit). On the other hand, in the flame resistant union fabric a percentage of the cellulosic fiber yarn (B) is not less than 30%, and preferably not less than 40% (lower limit), and it is not more than 70%, and preferably not more than 60% (upper limit).

Of course, a total of the compound yarn (A) and the cellulosic

fiber yarn (B) is adjusted to be 100% by weight.

A percentage of the compound yarn (A) of less than the lower limit in the flame resistant union fabric fails to provide sufficient flame resistance, and on the other hand, a percentage exceeding the upper limit fails to fully exhibit special feature as a flame resistant fiber of the fiber yarn (B).

Reasons that a flame resistant fiber union fabric of the present invention represents high flame resistance of class M1 in NF P 92-503 combustion test are not yet certain, but for example, following reasons can be conceivable.

- (1) Use of compound yarn (A) that cannot easily be elongated under temperatures of 100 degree C to 500 degrees C during heater-heating of combustion test suppresses contraction of the fabric by heat, and promotes carbonization at the time of contact to a flame of a heater to improve flame resistance.
- (2) Especially, mixing of fibers having thermal decomposition temperatures higher than that of the halogen-containing fiber, such as cotton, rayon, and aramid fibers, suppresses calorific power at the time of contact to a flame of a heater.

20

25

30

5

10

15

#### EXAMPLE

(Flame resistance examination)

Evaluation of flame resistance of union fabrics was performed according to French NF P 92-503 method. The French NF P 92-503 combustion test method will be briefly described. Examined fabric is held horizontally inclined by 30 degrees, an electric heater with 500 W is brought close to the fabric, and contact with a burner flame is carried out for 5 seconds at each timing of 20 seconds, 45 seconds, 75 seconds, 105 seconds, 135 seconds, and 165 seconds after heater heating starts. Flame resistance is judged by a number of seconds

in which a flame remains burning, and a distance of charring. This examination is a very severe combustion test in which contact with a burner flame is carried out simultaneously with heating by an electric heater.

Combustion of a union fabric was carried out in four directions of: warp surface side, warp reverse side, weft surface side, and weft reverse side. Judgment was performed according to following NF P 92-507 criterion.

Acceptance criterion

10 M1: All flame-remaining periods in 4 directions are not more than 5 seconds

M2: In examination in four directions, at least one sheet has a flame-remaining period exceeding 5 seconds, and an average distance of charring of not more than 35 cm

15 M3: In examination in four directions, at least one sheet has a flame-remaining period exceeding 5 seconds, and an average distance of charring of not more than 60 cm

(Measurement of elongation percentage)

Using SSC150 (manufactured by Seiko Instruments Inc.), a sample length to the original sample length was measured when a testing temperature was raised from 100 degrees C to 500 degrees C in a rate of 100-degree C/minute under a fixed load of 300 mg/metric count of No. 17. An elongation percentage is defined as a ratio of a difference between a sample length at the time of a maximum elongation at 100 degrees C to 500 degrees C, and an original sample length to an original sample length.

### Manufacturing Example 1

20

25

(Manufacture of a compound yarn of a halogen-containing flame 30 resistant fiber and cotton)

Acrylonitrile 52 parts, vinylidene chloride 46.8 parts, and sodium styrenesulfonate 1.2 parts were copolymerized to obtain an acrylic based copolymer. The obtained acrylic based copolymer was dissolved in acetone to obtain a solution with a concentration of 30%. Antimony trioxide 50 parts was added to the obtained copolymer 100 parts to prepare a spinning solution. The obtained spinning solution was extruded into an aqueous solution of acetone with a concentration of 38% at 25-degree C using a nozzle having 0.07 mm of pore size, and 33000 numbers of holes, and then after washing with water the obtained filaments were dried for 8 minutes at 120 degrees C. Then the obtained filaments were drawn 3 times at 150 degrees C, and subsequently heat-treated for 30 seconds at 175 degrees C to obtain a halogen-containing flame resistant fiber having a size of a fiber of 3 dtex. A finishing oil for spinning (manufactured by TAKEMOTO OIL & FAT CO., LTD.) were given to the obtained halogen-containing flame resistant fiber, textured to form crimps, and subsequently cut into Subsequently, the cut halogen-containing flame length of 38 mm. resistant fiber 80 parts and cotton 20 parts were mixed in a state of raw fiber so as to be a total of 100 parts to manufacture a spun yarn having a metric count of No. 17. Table 1 shows elongation percentage of obtained compound yarn.

## Manufacturing Example 2

5

10

15

20

25

30

(Manufacture of a compound yarn of a halogen-containing flame resistant fiber and cotton)

Except for having mixed cotton 30 parts to the halogen-containing flame resistant fiber 70 parts, a similar method as in Manufacturing Example 1 was repeated to manufacture a compound yarn and then a spun yarn having a metric count of No. 17. Table 1 shows elongation percentage of obtained compound yarn.

Manufacturing Example 3

(Manufacture of a compound yarn of the halogen-containing flame resistant fiber and cotton)

Except for having mixed a cotton 40 parts to the halogen-containing flame resistant fiber 60 parts, a similar method as in Manufacturing Example 1 was repeated to manufacture a compound yarn and then a spun yarn having a metric count of No. 17. Table 1 shows elongation percentage of obtained compound yarn.

10

15

25

5

Manufacturing Example 4

(Manufacture of a compound yarn of the halogen-containing flame resistant fiber and rayon)

Except for having mixed a rayon 20 parts to the halogen-containing flame resistant fiber 80 parts, a similar method as in Manufacturing Example 1 was repeated to manufacture a compound yarn and then a spun yarn having a metric count of No. 17. Table 1 shows elongation percentage of obtained compound yarn.

# 20 Manufacturing Example 5

(Manufacture of a compound yarn of halogen-containing flame resistant fiber and rayon)

Except for having mixed a rayon 30 parts to the halogen-containing flame resistant fiber 70 parts, a similar method as in Manufacturing Example 1 was repeated to manufacture a compound yarn and then a spun yarn having a metric count of No. 17. Table 1 shows elongation percentage of obtained compound yarn.

## Manufacturing Example 6

30 (Manufacture of a compound yarn of halogen-containing flame resistant

fiber and rayon)

5

10

15

20

Except for having mixed a rayon 40 parts to the halogen-containing flame resistant fiber 60 parts, a similar method as in Manufacturing Example 1 was repeated to manufacture a compound yarn and then a spun yarn having a metric count of No. 17. Table 1 shows elongation percentage of obtained compound yarn.

Comparative Manufacturing Example 1

(Manufacture of a halogen-containing flame resistant fiber)

A halogen-containing flame resistant fiber was manufactured in a same manner as in Manufacturing Example 1, and a spun yarn having a metric count of No. 17 was obtained without mixing cellulosic fiber. Table 1 shows elongation percentage of obtained compound yarn.

Examples 1 to 6

(Manufacture of union fabrics)

Using a spun yarn of cotton with a metric count of No. 51 (percentage of the warp yarn 55%) as a warp yarn with a density of 155 units/2.54 cm (1 inch), compound spun yarns manufactured in the Manufacturing Examples 1 to 6 were woven with a density of 42 units/2.54 cm (1 inch) (percentage of the weft yarn 45%) as weft yarns into union fabrics having a 5 harness satin weave. The obtained union fabrics were evaluated for flame resistance. Table 1 shows results.

Comparative Example 1

(Manufacture of union fabrics)

25 Except for having used a spun yarn manufactured in the Comparative Manufacturing Example 1 as a weft yarn, union fabrics of 5 harness satin weave were manufactured in a same manner as in Examples 1 to 6. The obtained union fabric was evaluated for flame resistance. Table 1 shows results.

Table 1

5

10

	Compound yarn (A)					
EXAMPL E Number	Antimony content in Halogen-containi ng fiber (a-1) (part)	Other fiber (a-2)	Mixture ratio (a-1)/(a -2)	Elon gati on perc enta ge (%)	Mixture ratio of compound yarn (A) / cellulosic fiber yarn (B) in a union fabric	Flame resistance
1	50	Cotton	80/20	0	45/55	M1
2	50	Cotton	70/30	0	45/55	M1
3	50	Cotton	60/40	0	45/55	M1
4	50	Rayon	80/20	0	45/55	M1
5	50	Rayon	70/30	0	45/55	M1
6	50	Rayon	60/40	0	45/55	M1
Compar ative Exampl e	5 <b>ọ</b>	-	100/0	35	45/55	M2

As is clear with reference to Table 1, compound yarns (A) in Manufacturing Example 1, 2, or 3 using the halogen-containing flame resistant fiber including antimony trioxide as a flame resistant agent and cotton have 0% of elongation percentage at 500 degrees C. And combustion test results of union fabrics in Example 1, 2, or 3 manufactured using the compound yarns (A) and spun yarn (B) of cotton has class M1, showing high flame resistance. Also in Examples 4, 5, or 6 using the rayon as a cellulosic fiber, combustion test results

have class M1 to show high flame resistance.

On the other hand, the spun yarn using only a halogen-containing flame resistant fiber manufactured by the Comparative Manufacturing Example 1, an elongation percentage at a temperature of 500 degrees C shows 35%. The union fabric in Comparative Example 1 manufactured using this compound yarn and a spun yarn of cotton has flame resistance inferior to that of union fabrics obtained in Examples 1 to 6, showing class M2.

As mentioned above, it may be understood that a union fabric consisting of a compound yarn obtained by compounding a halogen-containing flame resistant fiber including antimony trioxide and an other fiber, and a cellulosic fiber yarn can give a fabric having high flame resistance classified into class M1.

#### INDUSTRIAL APPLICABILITY

Since a flame resistant union fabric of the present invention is a union fabric having high degree of flame resistance that may passes class M1 of NF P 92-503 combustion test in France, it can develop high flame resistance also in union fabrics, such as jacquard, dobby, and satin weave.

20

5

10

15

25